

NAVAL ORDNANCE LABORATORY CORONA

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ABSTRACT

The hydrated strontium borate, veatchite (SrO·3B₂O₃·2H₂O), and its calcium isomorph (CaO·3B₂O₃·2H₂O) have been prepared by hydrothermal synthesis at 260°C. Veatchite was prepared by the reaction of Sr(OH)₂ and H₃BO₃ solution and also by the conversion of tunellite (SrO·3B₂O₃·4H₂O) in H₃BO₃ solution at 260°C. The calcium isomorph was prepared in a similar manner by the reaction of Ca(OH)₂ and H₃BO₃ solution and also by the conversion of gowerite (CaO·3B₂O₃·5H₂O) in H₃BO₃ solution at 260°C. The observed densities of the synthetic veatchite and the calcium isomorph, as determined with a pycnometer and using water as the liquid, were 2.77 ± 0.04 and 2.31 ± 0.02 g/respectively, for 0.5-g samples of fine crystals. The highest and lowest indices of refraction (white light) for the synthetic veatchite and its calcium isomorph were determined to be 1.546 and 1.619 ± 0.003, and 1.555 and 1.620 ± 0.002, respectively.

FOREWORD

The Naval Ordnance Laboratory Corona is making a continuing study of the chemistry and synthesis of crystals of ferroelectric colemanite (2CaO·3B₂O₃·5H₂O), its strontium isomorph (2SrO·3B₂O₃·5H₂O), and other related calcium and strontium borate hydrates of potential use in ferroelectric devices. As part of this study, the chemistry and methods of synthesis of veatchite (SrO·3B₂O₃·2H₂O) and its calcium isomorph (CaO·3B₂O₃·2H₂O) are reported herein. The dielectric, ferroelectric, and pyroelectric properties of the materials synthesized will be reported later. All the above work is authorized by ONR Contract PO 1-0008 NR 048-119.

C. J. HUMPHREYS
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INTRODUCTION

The synthesis of colemanite single crystals in 1959 and the preparation of deuterated colemanite in 1961 have been reported in NOLC Technical Memorandums (Refs. 1, 2, 3), and in 1962 a comparison of the ferroelectric and pyroelectric properties of mineral and synthetic colemanite was made (Ref. 4). The synthesis of the hydrated calcium borates was then investigated further to improve the quality and size of the crystals produced, and a limited study was made of the hydrothermal reactions that produce hydrated calcium and strontium borates and the transformation these borates undergo with increased temperature.

The work reported in this document is only a small part of the overall investigation of the calcium and strontium borate hydrates that might prove of interest to those people who are concerned with ferroelectric devices. Since, to the author's knowledge, there has been no previous laboratory synthesis of the mineral veatchite (SrO·3B₂O₃·2H₂O), and no mention of the existence of a calcium isomorph (CaO·3B₂O₃·2H₂O), this work is considered sufficiently important to be reported separately from the results of the entire investigation, which will appear in a report now in process of publication (Ref. 5).

PREPARATION OF SYNTHETIC VEATCHITE

It was found that synthetic veatchite could be prepared by two methods. By the first method, strontium hydroxide is reacted with boric acid in aqueous solution at 260°C for 64 hours. By the second method, the veatchite is prepared by the transformation of synthetic "tunellite" (SrO·3B₂O₃·4H₂O) in boric acid solution at 260°C for 40 hours. The second method produces the best quality of crystals.

Method I. Reaction of $Sr(OH)_2$ and H_3BO_3 . In this method and also in Method II, the reaction is carried out in a sealed, stainless steel bomb, which has a capacity of 76 ml and is equipped with a platinum liner. The platinum liner is not absolutely necessary, but it serves to reduce the contamination of the product. The reaction mixture consists of 8.50 ml H_2O (CO₂ free), 1.42 g anhydrous SrO, and 7.30 g boric acid

crystals. The reaction is completed within 64 hours at 260°C to form veatchite.

Method II. Transformation of tunellite (SrO·3B₂O₃·4H₂O) in boric acid solution. The reaction mixture consists of 22.6 ml H₂O (CO₂ free), 1.89 g synthetic tunellite, and 4.52 g boric acid crystals. The reaction is completed within 40 hours at 260°C to form veatchite. In the absence of boric acid, this reaction does not produce veatchite.

Table 1 gives the X-ray diffraction analysis for both the synthetic veatchite produced by the above methods and the calcium isomorph of veatchite discussed below. For comparison purposes, X-ray data are presented from investigations of the mineral veatchite by others (Refs. 6, 7, 8).

PREPARATION OF THE CALCIUM ISOMORPH OF VEATCHITE

Reactions similar to those described above produce the calcium isomorph (CaO·3B₂O₃·2H₂O). The reactions are identical, except that for Method I an equivalent amount of Ca(OH)₂ is substituted for the SrO, and in Method II gowerite (CaO·3B₂O₃·5H₂O) is substituted for tunellite. The reaction time is reduced to 40 hours at 260°C in the formation of the calcium compound, since longer reaction time favors the formation of other reaction products. See Table 1 for the X-ray diffraction data for the synthetic calcium isomorph produced by these reactions. Details of other reaction times and resultant products will be published in the forthcoming report on the entire investigation (Ref. 5).

This isomorph can also be produced by the conversion reaction of synthetic ginorite (2CaO·7B₂O₃·8H₂O) in boric acid solution in a manner identical with that given for the transformation of gowerite. In the presence of boric acid solution of the strength used in the above reactions, gowerite is completely transformed to ginorite at 170°C and to the veatchite isomorph at 260°C.

The compound CaO·3B₂O₃·2H₂O is also one component of the reaction product resulting when a mixture containing 0.20 g colemanite and 1.00 g priceite is reacted for 5 days at 200°C with 50 g of 20% boric acid solution in a 76-ml bomb. The second component of this reaction product will be discussed in the report cited in Ref. 5.

DENSITY AND REFRACTIVE INDEX OF SYNTHETIC VEATCHITE AND THE CALCIUM ISOMORPH

Two determinations of density were made with a pycnometer on separate samples of the synthetic veatchite crystals, using water as the liquid. The values obtained for the two specimens (approximately 0.5 g each) of fine crystals were 2.73 and 2.80 g/cm³. This compares favorably with the value of 2.78 \pm 0.03 reported by Clark, Mrose, Perloff, and Burley (Ref. 6).

Two determinations of density of the synthetic $CaO \cdot 3B_2O_3 \cdot 2H_2O$ were also made by the pycnometer method. The values obtained for the two specimens (approximately 0.5 g each) of fine crystals were 2.29 and 2.32 g/cm³.

The highest and lowest refractive indices were determined for the synthetic veatchite and $CaO \cdot 3B_2O_3 \cdot 2H_2O$ by using the immersion method and white light. The values for synthetic veatchite are 1.546 and 1.619 (\pm 0.003), and for the calcium isomorph, 1.555 and 1.620 (\pm 0.002).

TABLE 1. X-Ray Powder Data for Synthetic Veatchite, the Calcium Isomorph of Veatchite, and Mineral Veatchite

1	1	1	.*																		ł
	loff,	lated)	hk1	200	020	011	111	111	400	211	220	211	311	320	311	411	420	411	009	511	~ ~
	Clark, Mrose, Perloff, Burley, 1959	(calculated)	d_{hkl}	10.40	5.870	5.775	5.609	5.521	5.199	(5.115)	5.112	4.985	4.505	4.480	4.372	(3.924	(3.892	3.807	3,466	3.425	10001
tchite	Clark, M Burl	(measured)	I d _{hk1}	100 10.5		7 2 44				6 5.12			2 4.51		2 4.37	4 3.92		3 3,81	20 3.47		
Mineral Veatchite	Kramer and Allen, 1956	(measured)	I d _{hkl}	10 10.53		1 5 63))	-		3 5.22					1 4.20	1 3.93	•		10 3.47	,	
	Stewart, Chalmers, and Phillips, 1954	(measured)	I* d _{bkl}	vs ¹ 10.3		7 7 7				m 5.13				w 4.47			m 3.88		w 3.48		
Calcium Isomorph of Veatchite—	Parkerson, 1963	(measured)	I d _{hkl}	100 10.47		7 7 7	•	1 5.27		4 5.15	1 4.92	1 4.79	1 4.48	1 4.37	1 4.11	>1 4.02	>1 3.77	1 3.69	18 3.42		
Synthetic Veatchite—	Parkerson, 1963	(measured)	I d _{bkl}	100 10.5		ע		1 5.43		4 5.15		1 4.79	1 4.52	1 4.46	1 4.13	2 3.88	2 3.83	1 3.67	22 3.48		

NOTE: Footnotes are at end of table.

TABLE 1. (contd.)

Synthetic Veatchite—	Calcium Isomorph of Veatchite—		Mineral Veatchite	atchite		
Parkerson, 1963	Parkerson, 1963	Stewart, Chalmers, and Phillips, 1954	Kramer and Allen, 1956	Clark, Mr Burle	Clark, Mrose, Perloff, Burley, 1959	off,
(measured)	(measured)	(measured)	(measured)	(measured)	(calculated)	ited)
I dhkl	I d _{bk1}	I* d _{hk1}	I dhkl	I d _{hkl}	dhkl	hkl
			:		3.394	520
9 3.37:				3 3.37	3.370	031
(2		٠.	(3.336	131
3,33	14 3.28	vs 3.33	2 3.32	35. 3.32	3.328	511
					3.318	131
1 3 23	.1 3 22	3.20		3 3 33	3.316	002
) •	11:0	3		77.0	3.192	702 703
>1 3.15	>1 3.14				3.190	231
	3 3.09				3.128	202
·	3 3.05		• ,		3.053	331
1 3.02					(3.013	611
	>1 2.995	wb 2.99		3 _b 3.00	3.010	331
1 2.986	:		•)	(2.985	970
	>1 2.947				(2.935	040
1 2.938			,	2 2.936	(2.933	611
					5.906	140
	3 2,882				2.887	022
8 2.864		m 2.87	3 2.88	9 2,865	2.872	122
					2.851	431
					ن	-

NOTE: Footnotes are at end of table.

FABLE 1. (contd.)

Synthetic	Calcium Isomorph		Mineral Veatchite	atchite		
Veatchite— Parkerson, 1963	or veatchite— Parkerson, 1963	Stewart, Chalmers, and Phillips, 1954	Kramer and Allen, 1956	Clark, Mrose Burley,	Clark, Mrose, Perloff, Burley, 1959	loff,
(measured)	(measured)	(measured)	(measured)	(measured)	(calculated)	ated)
I dhkl.	I d _{hk} l	I* dhkl	I dhkl	I d _{hkl}	dhkl	hkl
	2 2.846				2.848	122
					2.841	402
2 2.820	5 2.812				2.825	240
2 2.803	• • • • • • • • • • • • • • • • • • • •		j.	1 2.798	908.2	431
					(2.804	222
2 2,778		vvw 2.77	2.76	1 2.763	2.761	222
				•	2.753	402
>1 2.704		vvw 2.70	2.67	1 2.704	2,703	340
			······································		2.695	322
>1 2.673					2.675	711
	1 2.642		•		2.651	720
					2.642	531
					2.637	322
					2.610	711
25 2.605	24 2.569	s ² 2.60	10 2.61	25 2.600	2.600	800
•					2.596	531
	3 2.526			1 2.564	2.558	422
		vw 2.53			2.556	440
1 2.486	1 2.486			1 2.495	2.492	422
1 2.434	1 2.453				2.439	<u>6</u> 02
[[[(Contd.	d.)

NOTE: Footnotes are at end of table.

·TABLE 1. (contd.)

Synthetic	Calcium Isomorph				
Veatchite-	of Veatchite.		Mineral Veatchite	atchite	·. . ·
Parkerson, 1963	Parkerson, 1963	Stewart, Chalmers, and Phillips, 1954	Kramer and Allen, 1956	Clark, Mrose, Burley, 1	ose, Perloff, y, 1959
(measured)	(measured)	(measured)	(measured).	(measured)	(calculated)
I d _{bkl}	I d _{hk1}	I* dhkl	I d _{hkl}	I d _{hkl}	d _{hkl} hkl
:					2,438 631
3 2.399		m 2,39	. 1 . 2.40	6 2,398	54
. (:	•		
1 2.372	•			••	2,377 820
			:		2.356 602
	•				2.344 811
	1 2.274		· ·		2,339 522
1 2,252	1 2.252	,	:	2.245	
5 2.204	1 2,210	. m 2.20	1 2.21	3 2,204	
4 2.164	>1 2.174		1 2.17	2 2.171	
4 2,154	3 2.154	w 2,151	I 2,16	2 2,155	
2 2.130	1 2,125			2 2,115	
2 .2.120		.m. 2.082	9 2.08	6 2.079	
		m 2.037		4 2.045	
5 2.043	4 2.047	vw 1.996		4 2.029	
5 2.030	2 2,021			3b** 1.958	
4 2.009	3 1.996	m 1.936	1 1.924	3 1.925	
1 1.980				2 1.876	
1 1.959	2 · 1.967	vw 1.863		2 1.854	
					1 (6224)

NOTE: Footnotes are at end of table.

ABLE 1, (contd.)

	•				
-Synthetic	Calcium Isomorph		Mineral Vestchite	atchite	
Veatchite-	of Veatchite-			יייייייייייייייייייייייייייייייייייייי	
Parkerson,	Parkerson,	Stewart, Chalmers,	Kramer and	Clark, Mr	Clark, Mrose, Perloff,
1963	1963	and Phillips, 1954	Allen; 1956	Burle	Burley, 1959
(measured)	(measured)	(measured)	(measured)	(measured)	(calculated)
I d _{hkl}	I d _{hkl}	I* d _{hkl}	I d _{hkl}	I d _{hkl}	d _{hkl} hkl
2 1.943		vvw .1.836	1 1.833	2 : 1.828	
2 1.928	•	vw 1,801		4 1.784	
1 1,905		wb 1.761	2 1.735	1. 1.730	
1 1.868	1 1.854			1 1.700	
1 1.843	1 1.833	vvw 1.676		1 1.680	
1 1.836	1 1.819	vvw 1.653		3 1.660	
1 1,783	1 1.783				
i 1.763	1 1.760				÷
1 1.732					
>1 1.714	1 1.708		•		
1 1.674	,				
2 1.657	1 1.649				

* For the Stewart, Chalmers, and Phillips data, the order of decreasing intensities is: vs1, vs2, s³, mw, vw, vvw.

* b indicates broadline.

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The hydrated strontium borate, veatchite (SrO·3B ₂ O ₃ ·2H ₂ O), and its calcium isomorph (CaO·3B ₂ O ₃ ·2H ₂ O) have been prepared by hydrothermal synthesis at 260°C. Veatchite was prepared by the reaction of Sr(OH) ₂ and H ₃ BO ₃ solution and also by the conversion of tunellite (SrO·3B ₂ O ₃ ·4H ₂ O) in H ₃ BO ₃ solution at 260°C. The calcium isomorph was prepared in a similar manner. The observed densities of the synthetic veatchite and the calcium isomorph, as determined with a pycnometer and using water as the liquid, were 2.77 ± 0.04 and 2.31 ± 0.02 g/cm ³ respectively, for 0.5-g samples of fine	R: PO 1-0008 NR 048-119	3.	ONR: PO 1-0006 NR 048-119
103,	This card is UNCLASSIFIED	crystals. The highest and lowest indices of refraction (white light) for the synthetic veatchite and its calcium isomorph were determined to be 1.546 and 1.619 ± 0.003, and 1.555 and 1.620 ± 0.002, respectively.	This card is UNCLASSIFIED
Naval Ordnance Laboratory Corona. (NOLC Report 583) 1. HYDROTHERMAL SYNTHESIS OF VEATCHITE AND 2. ITS CALCIUM ISOMORPH, by C. R. Parkerson, Research Department, 1 June 1963, 16 pp. UNCLASSIFIED 1.			1. Calcium borate— Synthesis 2. Strontium borate— Synthesis 3. Veatchite—Synthesis 11. Parkerson, C. R.
The hydrated strontium borate, veatchite (SrO·3B ₂ O ₃ . 2H ₂ O), and its calcium isomorph (CaO·3B ₂ O ₃ . 2H ₂ O) have been prepared by hydrothermal synthesis at 260°C. Veatchite was prepared by the reaction of Sr(OH) ₂ and H ₃ BO ₃ solution and also by the conversion of tunellite (SrO·3B ₂ O ₃ ·4H ₂ O) in H ₃ BO ₃ solution at 260°C. The calcium isomorph was prepared in a similar manner. The observed densities of the synthetic veatchite and the calcium isomorph, as determined with a pycnometer and using water as the liquid, were 2.77 ± 0.04 and 2.31 ± 0.02 g/cm³, respectively, for 0.5-g samples of fine prystals. The bishest and lowest indices of refraction	DOR: PO 1-0008 NR 048-119	ave ave	ONR: PO 1-0008 NR 048-119
3,	This card is UNCLASSIFIED	(white light) for the synthetic veatchite and its calcium isomorph were determined to be 1.546 and 1.619 \pm 0.003, and 1.555 and 1.620 \pm 0.002, respectively.	This card is UNCLASSIFIED